

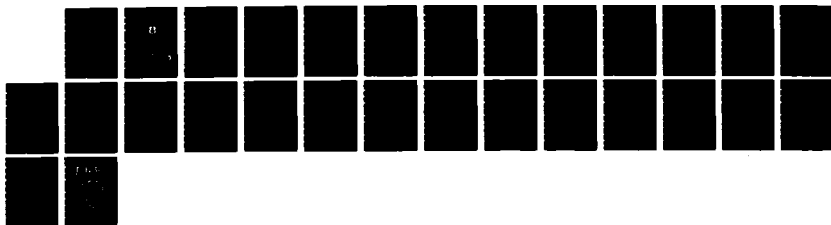
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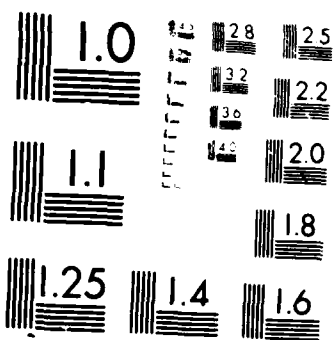
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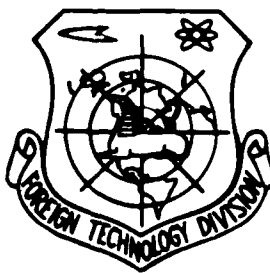
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(Selected Articles)



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FTD-ID(RS)T-0846-87

11 September 1987

MICROFICHE NR: FTD-87-C-000740

HIGH-MOLECULAR COMPOUNDS (Selected Articles)

English pages: 20

Source: Vysokomolekulyarnyye Soyedineniya, Seriya B,
Kratkiye Soobshcheniya, Vol. 14, Nr. 10,
October 1972, pp. 736-740; 757-760

Country of origin: USSR

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Foreign page numbers occur in the English text and may be found anywhere along the left margin of the page as in this example:

In them occurs the state named "night blindness" - hemeralopia, which, according to the current point of view, is a result of damage of the rod-shaped apparatus of the eye.

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However, in recent years it has been shown that with the hereditary pigment degenerations in animals the biochemical changes are observed in all cellular elements of the retina.

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U. S. BOARD ON GEOGRAPHIC NAMES transliteration SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А	<i>А а</i>	A, a	Р	<i>Р р</i>	R, r
Б	<i>Б б</i>	B, b	С	<i>С с</i>	S, s
В	<i>В в</i>	V, v	Т	<i>Т т</i>	T, t
Г	<i>Г г</i>	G, g	У	<i>У у</i>	U, u
Д	<i>Д д</i>	D, d	Ф	<i>Ф ф</i>	F, f
Е	<i>Е е</i>	Ye, yə; E, e*	Х	<i>Х х</i>	Kh, kh
Ж	<i>Ж ж</i>	Zh, zh	Ц	<i>Ц ц</i>	Ts, ts
З	<i>З з</i>	Z, z	Ч	<i>Ч ч</i>	Ch, ch
И	<i>И и</i>	I, i	Ш	<i>Ш ш</i>	Sh, sh
Я	<i>Я я</i>	Y, y	Щ	<i>Щ щ</i>	Shch, shch
К	<i>К к</i>	K, k	Ъ	<i>Ъ ъ</i>	"
Л	<i>Л л</i>	L, l	Ы	<i>Ы ы</i>	Y, y
М	<i>М м</i>	M, m	Ь	<i>Ь ь</i>	'
Н	<i>Н н</i>	N, n	Э	<i>Э э</i>	E, e
О	<i>О о</i>	O, o	Ю	<i>Ю ю</i>	Yu, yu
П	<i>П п</i>	P, p	Я	<i>Я я</i>	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ѐ in Russian, transliterate as yě or ě.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh ⁻¹
cos	cos	ch	cosh	arc ch	cosh ⁻¹
tg	tan	th	tanh	arc th	tanh ⁻¹
ctg	cot	cth	coth	arc cth	coth ⁻¹
sec	sec	sch	sech	arc sch	sech ⁻¹
cosec	csc	csch	csch	arc csch	csch ⁻¹

Russian English

rot curl
lg log

GRAPHICS DISCLAIMER

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STABILIZATION OF ACRYLIC POLYMERS WITH DIFFERENT FUNCTIONAL GROUPS.

A. A. Berlin, G. N. Shvareva, Yu. A. Makarov, M. A. Zvereva.

In work [1] based on example of polymethyl methacrylate (PMMA [PMMA-polymethyl methacrylate]) were given results of experiments on stabilization of polyacrylates during thermo-oxidizing destruction by introduction to polymer chain of thioalkyl-acrylic.

Represented interest emission by proposed method of stabilization of polymers, which contain different functional groups, for example amide, carboxyl, hydroxyl.

As subject of investigation were taken copolymers of butylmethacrylate (BMA) with methacrylamide (AMK), methacrylic acid (MAK) and ethylene-glycolmethacrylate (MEG), copolymer of methylmethacrylate (MMA) and MAK and polybutylmetacrylate. The content of MAK and ethylene-glycolmethacrylate in the copolymers composed 8.74 mol.%, AMK - 11.2 mol.%. 1

Experimental part.

Monomers, utilized in work, according to data of chromatographic analysis, had content of base material: MMA - 99.9, BMA - 99.6 - MAK - 99.8 MEG - 99.4 ethylthioethylmethacrylate (ETEMA) - 99.7%. AMK had

melting point of 108.5°.

Polymers were obtained by method of varnish polymerization in medium of isopropyl or butyl alcohol with dinitrile of azoisobutyric acid (DAK). Samples for the tests re-precipitated from acetone by the aqueous solution of aluminopotassium alum or by water.

DAK re-precipitated from isopropyl alcohol, melting point of 105°.

Acetone, isopropyl and butyl alcohols had qualification analytically pure (analytical grade (reagent)). The thermostability of samples was estimated by thermogravimetric method at 285° on the torsion weights VT-200.

Samples had intrinsic viscosities: copolymer BMA and AMK - 0.37, copolymer BMA and MAK - 0.34 BMA and ETEMA - 0.29, polybutylmethacrylate - 0.284 dl/g.

Results and their discussion.

Fig. 1 gives curves of thermo-oxidizing destruction of copolymers BMA, AMK and ETEMA, taken in different relationships/ratios.

Fig. 2 depicts dependence of thermostability, expressed by reciprocal value of decrease in weight $1/\Delta P$ by samples at 285° for 1

hour, from content in copolymer of ETEMA (curve 1). As in the case of stabilization of PMMA [1], the dependence of thermostability on the content in the copolymer of thioalkyl-acrylate carries, the extreme character, caused by the presence of the passing simultaneously two processes - inhibited oxidation of copolymer and decomposition of sulfidecontaining fragments.

Copolymer, which contains 5 wt.% ETEMA from sum of BMA and AMK, has maximum thermostability.

Fig. 3 shows change in content of sulfur in polymeric remainder during thermo-oxidizing destruction of copolymer of BMA, AMK and ETEMA, and also change of decrease in weight by sample in time.

As is evident, with decrease content of sulfur in polymeric remainder/residue respectively is increased decrease in weight by sample.

During stabilization of copolymer of BMA and MAK expected effect was not obtained, about which Fig. 4a, where curves of process of thermo-oxidizing decomposition of this copolymer are given, testifies.

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FP
The absence of the effect of stabilization is connected, apparently, with the presence in copolymer of the carboxyl groups, reaction with which of sulfur sulfide decontaminates the stabilizing effect of the latter (data of IR spectroscopy show a change in the intensity of absorption band).

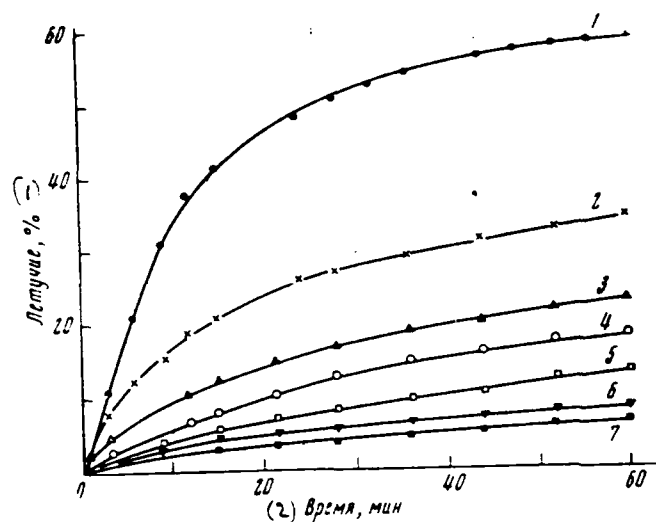


Fig. 1. Curves of thermo-oxidizing destruction of copolymers of BMA, AMK and ETEMA at 285° without ETEMA (1); 0.1 (2), 0.5 (3), 1 (4), 3 (5), 7 (6) and 5% ETEMA from sum BMA+AMK (7).

Key: (1). Volatile components. (2). Time, min.

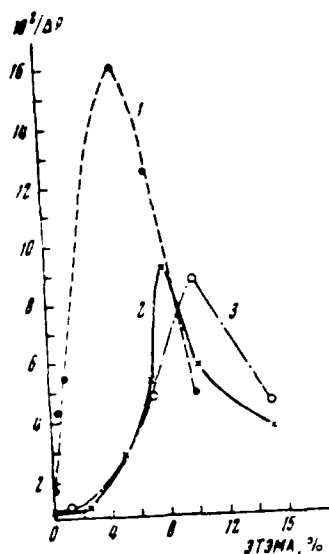


Fig. 2.

Fig. 2. Change in thermostability of copolymers from content of ETEMA. Copolymer BMA, AMK, ETEMA (1), BMA, MEG and ETEMA (2) and BMA and ETEMA (3).

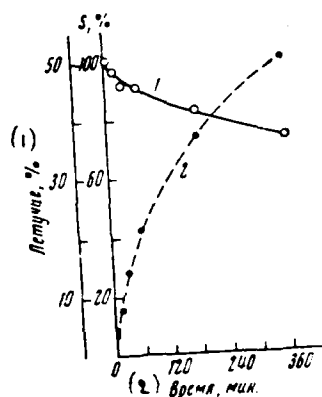


Fig. 3.

Fig. 3. Change in content of sulfur in polymeric remainder/residue (1) and decrease in weight (2) in process of thermo-oxidizing decomposition of copolymer BMA, AMK and ETEMA at 285°.

Key: (1). Volatile components. (2). Time, min.

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The confirmation of this is also the fact that the effect of stabilization of PMMA by copolymerizing methylmethacrylate with 0.35% ETEMA, shown in [1], was considerably lowered during the introduction to copolymer of MAK. Decrease in weight grew from 6.5 to 22% (Fig. 4b, curves 2 and 4).

Fig. 4c gives curves of thermooxidative destruction of polybutylmethacrylate and its copolymers BMA with ETEMA. As in the case of polybutylacrylate, which is easily depolymerized at temperatures higher than 200° with the almost quantitative exit of monomer, ETEMA considerably raises its stability - decrease in weight after 1 hour at 285° is lowered from 97 to 11% (curves 1 and 4).

In copolymer BMA, MEG and ETEMA also occurs increase in thermostability ~10 times during introduction of 8% thioalkyl-acrylate (Fig. 4d, curves 1 and 7).

During analysis of curves of oxidation destruction of polymers given above it is possible to note that in all cases extreme dependence of thermostability on content in copolymer of thioalkyl-acrylate (Fig. 2) is observed.

Value of optimal additive is greater, the lower temperature of vitrification of stabilized polymer.

Are given below temperatures of vitrification of investigated polymers determined by method of powder thermal mechanics, and in Fig. 5 - dependence of value of optimal additive ETEMA, which effectively inhibits thermo-oxidizing decomposition, from temperature of vitrification (T_v) of stabilized polymer.

(1) Полимер	ПММА	БМА и АМК ⁽²⁾	БМА и МЭГ ⁽²⁾	(3) полибутил- метакрилат
$T_v, ^{\circ}\text{C}$	104	42	26,5	19,5
(4) Оптимальная добавка ЭТЭМА к сомономерам, %	0,35	5	6	10

Key: (1). Polymer. (2). and. (3). polybutylmetacrylate. (4).
Optimal additive of ETEMA to comonomers, %.

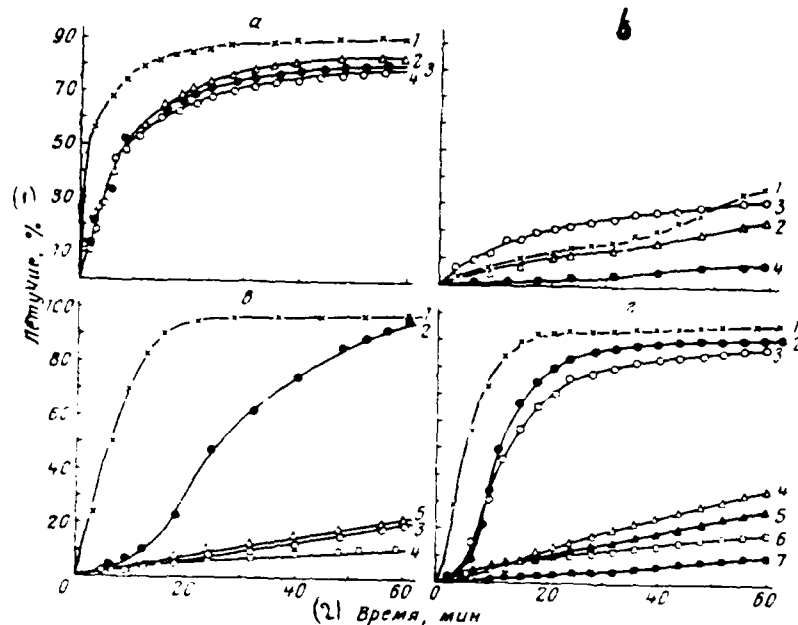


Fig. 4. Curves of process of thermo-oxidizing decomposition at 285°:
 a) copolymers BMA, MAK and ETEMA; quantity of ETEMA, wt. %: 1 - 0, 2 - 1, 3 - 5, 4 - 7; b) Δ PMMA; 2, 3 - copolymers MMA, MAK and ETEMA (0.5 and 5% ETEMA respectively); 4 - copolymer MMA and 0.35% ETEMA; c) copolymers BMA and ETEMA; quantity of ETEMA, wt. %: 1 - 0, 2 - 1, 3 - 3, 4 - 10, 5 - 15; d) copolymer BMA, MEG and ETEMA; content of ETEMA, wt. %: 1 - 0, 2 - 1, 3 - 3, 4 - 5, 5 - 15, 6 - 7, 7 - 8.

Key: (1). Volatile components. (2). Time, min.

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Process of chemical transformation of macromolecule (process of its decomposition) will pass more active, the higher mobility of segment. With reduction T , the probability of the primary recombination of free radicals in zone of decomposition decreases as a result of reduction in the viscosity of medium [2-4], therefore, the

lower T_c polymer, the greater the concentration of antioxidant is necessary for the suppression of the process of decomposition. If for the stabilization of PMMA, which has $T_c = 104^\circ$, is sufficient to have in the copolymer 0.35 wt.% ETEMA, then for the inhibition of the thermo-oxidizing decomposition of polybutylmetacrylate ($T_c = 19.5^\circ$) the value of additive increases/grows to 10%. The graphic dependence, given in Fig. 5, allows without the experimental selection in the first approximation, to determine the concentration necessary for the stabilization of sulfur-containing component T_c of the stabilized polymer.

It is necessary to note that effect of carboxyl group also is found in dependence on T_c polymer. Fig. 6 gives the curves of the thermo-oxidizing destruction of copolymer of BMA with optimal quantity of ETEMA (10%, curve 2) and copolymer BMA, MAK and ETEMA, whose optimal additive in accordance with the curve of Fig. 5 must be 5-5.5 wt.% (curve 1).

From comparison of curves of Fig. 6 and curves 2 and 4 Fig. 4b it is evident that in the case of polymer, which has lower T_c , effect of carboxyl-containing component is considerably stronger - difference in decrease in weight composes 69%, whereas for methylmethacrylate copolymers it is lowered to 16%.

Conclusions/derivations.

1. Possibility of stabilization of acrylic polymers, which contain functional groups (amide, hydroxyl), is shown by introduction to polymer chain of thioalkyl-acrylic.

2. It is established that presence in polymer of links, which contain carboxyl groups, considerably lowers stabilizing effect.

3. Is shown presence of dependence between temperature of vitrification T_g of polymer and optimal dosage of thioalkyl-acrylate, which effectively inhibits oxidation decomposition: with increase it respectively decreases.

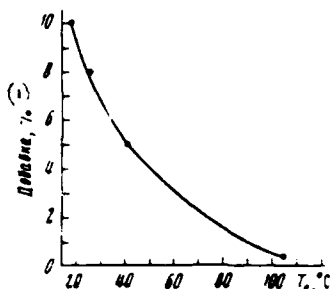


Fig. 5.

Fig. 5. Dependence of value of optimal additive of ETEMA from stabilized polymer.

Key: (1). Additive.

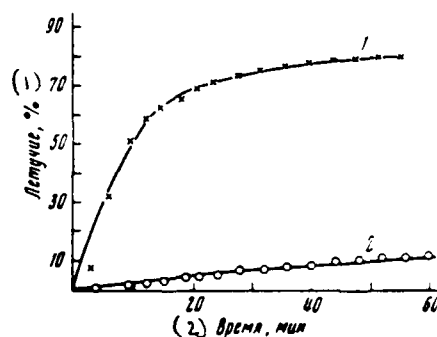


Fig. 6.

Fig. 6. Curves of thermo-oxidizing decomposition of copolymers BMA, MAK and ETEMA (1) and copolymers BMA and ETEMA (2) at 285°.

Key: (1). Volatile components. (2). Time, min.

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It was received by the editorial staff of 1 Feb. 1971.

Page 757.

PHOTOELASTIC EFFECT IN FILMS OF POLYVINYL CINNAMATE.

M. G. Vitovskaya, E. P. Astapenko, V. N. Tsvetkov.

In work [1] as a result of investigations of double refraction in flow of dilute solutions of fractions of polyvinylcinnamate (PVTs) it was shown that introduction to lateral groups of conjugated bonds considerably increases their equilibrium hardness. This property is developed in the high value of the optical anisotropy of monomer units in the macromolecules of PVTs. It is of interest to explain, in what measure the molecular peculiarities indicated are retained during the translation/conversion of macromolecules of PVTs from the dilute solutions into the conditions for the strong intermolecular interactions, which occur in the condensed (block) polymer. For this purpose in this work the study of the photoelasticity of films of PVTs in the temperature range, which covers the regions of the glassy and highly elastic states of polymer, was conducted.

Experimental data and their discussion.

Films for studying double refraction were prepared from unfractionated sample PVTs, $M_{SD} = 2,2 \cdot 10^5$, investigated in work [1]. Molding/formation of films was conducted from the solutions in the chloroform on the mercury and glass bases. The films, decanted on mercury, were opalesced, decanted on the glass - were transparent. Essential difference in the photoelastic properties of each films was

not observed, experimental points lie down on one dependence.

For each assigned temperature and tensile stress P were determined "equilibrium" values of double refraction Δn taking into account temporary/time dependences Δn . In the highly elastic state the double refraction (negative on the sign) was set in the films of PVTs virtually instantly and was constant in time. In the temperature range of the transition of dependence of Δn on the time τ they took the form of the relaxation curves $\Delta n = f(\tau)$.

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Dependences of equilibrium values Δn from appropriate stresses/voltages are depicted in Fig. 1 for series/number of temperatures. The rectilinear character of these dependences $\Delta n = f(P)$ makes it possible to describe the optical-mechanical state of polymer at an assigned temperature with the value of the coefficient of photoelasticity $\epsilon = \Delta n/P$, whose change with temperature $\epsilon = f(T)$ is represented in Fig. 2 (curve 1). In the experimental curve $\epsilon = f(T)$ distinctly is developed the transition of polyvinylcinnamate from glassy state to rubberlike, to which corresponds sharp change in the coefficients ϵ . Determined thus mean temperature of softening (vitrification) T , polyvinylcinnamate is equal to 85° . In the glassy state photoelastic effect of PVTs is positive on the sign, in the highly elastic - it is negative and has very high value ($\epsilon_{\text{el}} = -10,5 \cdot 10^{-10} \text{ cm}^2/\text{dyne}$), 2.5 times exceeding ϵ of polystyrene - one of the most optically anisothrone polymers ($\epsilon_{\text{el}} = -4 \cdot 10^{-10} \text{ cm}^2/\text{dyne}$).

Scatter of experimental points in curve $\epsilon=f(T)$ in high-temperature range ($T > T_f$) to a considerable degree is result of process of thermal structuring of films of PVTs. As a result it changes this polymer into the undissolved state, its softening temperature is raised and the value of coefficients ϵ in the highly elastic state decreases. Illustration is curve 2 (Fig. 2), which depicts dependence $\epsilon=f(T)$ for the film of PVTs, preliminarily sustained at 165° during 6 hour.

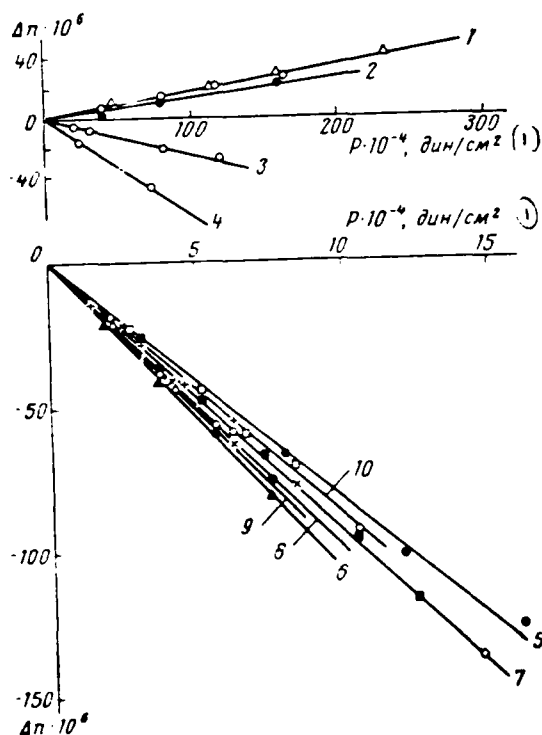


Fig. 1. Dependence of equilibrium double refraction of PVTs on constant stretching stress P . Temperature, $^{\circ}\text{C}$: 1 - 18, 22; 2 - 40; 3 - 61; 4 - 72; 5 - 85; 6 - 105; 7 - 108, 128, 143; 8 - 97, 120; 9 - 149, 154; 10 - 166.

Key: (1). dyn/cm^2 .

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Optically to the process of structuring corresponds decrease in the time of double refraction Δn (with constant stress P on the film). From Fig. 3 it is evident that the incidence/drop in effect Δn occurs earlier and with the greater speed, the higher the temperature of sample. The observed phenomena, apparently, are connected with the formation in the polymer of the intermolecular three-dimensional/space

grid, the denseness of cross-linkings of which is increased with temperature and annealing time of films.

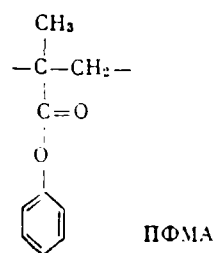
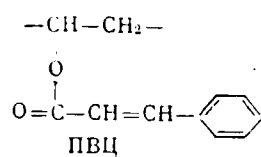
From values ϵ in highly elastic state optical anisotropy of static segment $\alpha_1 - \alpha_2$, was calculated according to formula [2]

$$\alpha_1 - \alpha_2 = \frac{45kT}{2\pi} \frac{n}{(n^2 + 2)^2} \epsilon_{\text{a.эл}}$$

Its average/mean value is equal to $\alpha_1 - \alpha_2 = -(310 \pm 10) \cdot 10^{-25} \text{ cm}^3$. The segmental anisotropy of the polymer chain is determined by [3] differences in the main polarizability of its monomer unit $a_{\parallel} - a_{\perp}$ and by its equilibrium hardness - number S of links in the segment

$$\alpha_1 - \alpha_2 = S(a_{\parallel} - a_{\perp})$$

If we consider that equilibrium hardnesses of molecular chains in highly elastic state of polymer and in its solution coincide, then, by substituting the value of $S=12$ [1], we will obtain for anisotropy of link of PVTs high value $a_{\parallel} - a_{\perp} = -26 \cdot 10^{-25} \text{ cm}^3$. It is known for [3] that the optical anisotropy of the polymers, which contain aromatic cycles in the lateral group, is negative and is determined in essence by the anisotropy of these cycles. In this case its absolute value is less, the further from the main chain is arranged/located the cycle, i.e., the less is braked its rotation. Obtained anisotropy $a_{\parallel} - a_{\perp}$ for the macromolecules of PVTs in the mass it is expedient to compare with value $a_{\parallel} - a_{\perp}$ for polyphenylmethacrylate (PFMA) in the same state, since the structures of these polymers are similar



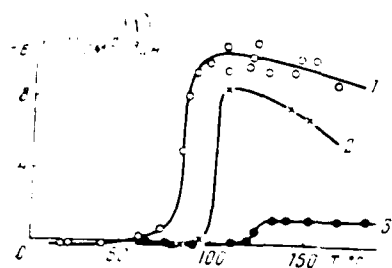


Fig. 2.

Fig. 2. Dependences ϵ of polyvinylcinnamate (1, 2) and polyphenylmethacrylate (3) on temperature.

Key: (1). cm^2/dyn .

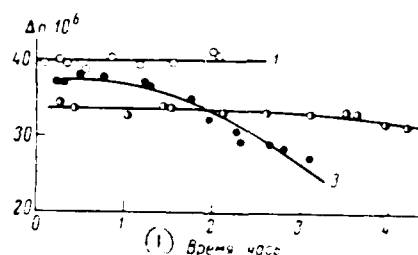


Fig. 3.

Fig. 3. Dependence of Δn on time (time was counted off from moment/torque of establishment of temperature) with constant P in films of PVTs with their thermal structuring. Temperature, $^{\circ}\text{C}$: 1 - 105, 2 - 120, 3 - 154; $P \cdot 10^4$, dyn/cm^2 : 1, 3 - 3.7; 2 - 3.3.

Key: (1). Time, hours.

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Temperature dependence ϵ for PFMA is presented to curved 3 (Fig. 2) [4]. To values ϵ in the highly elastic state corresponds anisotropy of segment PFMA $\alpha_1 - \alpha_2 = -43 \cdot 10^{-25} \text{ cm}^3$ the anisotropy of monomer unit $a_1 - a_2 = -6.2 \cdot 10^{-25} \text{ cm}^3$. In lateral groups of PVTs benzene ring is distant from the main chain in comparison with PFMA to the group $-\text{CH}=\text{CH}-$. It would be possible to expect that negative anisotropy $a_1 - a_2$ of monomer units of PVTs will be less than for PFMA. However, experiment gives the reverse: value Δ $\frac{(a_1 - a_2)_{\text{PVT}}}{(a_1 - a_2)_{\text{PFMA}}}$ four times exceeds $(a_1 - a_2)_{\text{PFMA}}$, which will qualitatively agree with the data,

obtained for the same polymers in solutions [1]. Consequently, as under the conditions of maximum dilution, the high value of the monomeric anisotropy of macromolecules of PVTs, observed in the mass, is caused by a strong increase of the retardation of rotation in the lateral groups of PVTs and by a high degree of their orientational order as a result of the introduction to them of groups $-\text{CH}=\text{CH}-$, which contain the conjugated carbon bonds. The fact that value $a - a_{\perp}$ in the block somewhat less than in the solution, can be connected with the specific effect of intermolecular interactions on the optical properties of polymeric molecules in the condensed state. One should note that the presence of coupling in the lateral radicals of PVTs, which so/such considerably raises their hardness, virtually does not affect the softening temperature (85°), which is lower than in PFMA (125°). Obviously, the absence of α -methyl group in the main chain of macromolecules contributes to the decrease in them of steric interference and to a reduction in the softening temperature. Let us note that a similar phenomenon is observed also during the comparison of softening temperatures of analogous polyesters/polyethers of acrylic and methacrylic series/numbers [4, 5].

Conclusions/derivations.

1. Photoelasticity of films of polyvinylcinnamate (PVTs) in range of temperatures of $18-170^{\circ}$ is investigated. Are determined the softening temperature ($T_s = 85^{\circ}$) and optical anisotropy of segment $\alpha_1 - \alpha_{\perp} = -310 \cdot 10^{-25} \text{ cm}^3$ of macromolecules of PVTs.

2. High value $\alpha - \alpha_1$ of macromolecules of PVTs in condensed (block) state, as in dilute solutions, it testifies about strong retardation of rotation and high degree of orientational order in lateral groups of PVTs as a result of presence in them of conjugated bonds.

3. At high temperatures ($T_D - T_c$) in films of PVTs process of thermal structuring occurs, as a result of which polymer changes into undissolved state.

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<u>ORGANIZATION</u>	<u>MICROFICHE</u>
A205 DMAHTC	1
A210 DMAAC	1
B344 DIA/RTS-2C	9
C043 USAMIA	1
C500 TRADOC	1
C509 BALLISTIC RES LAB	1
C510 R&T LABS/AVRADCOM	1
C513 ARADCOM	1
C535 AVRADCOM/TSARCOM	1
C539 TRASANA	1
C591 FSTC	4
C619 MIA REDSTONE	1
D008 NISC	1
E053 HQ USAF/INET	1
E404 AEDC/DOF	1
E408 AFWL	1
E410 AD/IND	1
E429 SD/IND	1
P005 DOE/ISA/DDI	1
P050 CIA/OCR/ADD/SD	2
AFIT/LDE	1
FTD	
CCN	1
NLA/PHS	1
LLNL/Code L-389	1
NASA/NST-44	1
NSA/1213/TDL	2
ASD/FTD/1Q LA	1

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